

* * * * * Welcome to STN International * * * * *

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NEWS 2 AUG 10 Time limit for inactive STN sessions doubles to 40 minutes
NEWS 3 AUG 18 COMPENDEX indexing changed for the Corporate Source (CS) field
NEWS 4 AUG 24 ENCOMPLIT/ENCOMPLIT2 reloaded and enhanced
NEWS 5 AUG 24 CA/CAPLUS enhanced with legal status information for U.S. patents
NEWS 6 SEP 09 50 Millionth Unique Chemical Substance Recorded in CAS REGISTRY
NEWS 7 SEP 11 WPIDS, WPINDEX, and WPIX now include Japanese FTERM thesaurus
NEWS 8 OCT 21 Derwent World Patents Index Coverage of Indian and Taiwanese Content Expanded
NEWS 9 OCT 21 Derwent World Patents Index enhanced with human translated claims for Chinese Applications and Utility Models
NEWS 10 NOV 23 Addition of SCAN format to selected STN databases
NEWS 11 NOV 23 Annual Reload of IFI Databases
NEWS 12 DEC 01 FRFULL Content and Search Enhancements
NEWS 13 DEC 01 DGENE, USGENE, and PCTGEN: new percent identity feature for sorting BLAST answer sets
NEWS 14 DEC 02 Derwent World Patent Index: Japanese FI-TERM thesaurus added
NEWS 15 DEC 02 PCTGEN enhanced with patent family and legal status display data from INPADOCDB
NEWS 16 DEC 02 USGENE: Enhanced coverage of bibliographic and sequence information
NEWS 17 DEC 21 New Indicator Identifies Multiple Basic Patent Records Containing Equivalent Chemical Indexing in CA/CAPLUS
NEWS 18 JAN 12 Match STN Content and Features to Your Information Needs, Quickly and Conveniently
NEWS 19 JAN 25 Annual Reload of MEDLINE database

NEWS EXPRESS MAY 26 09 CURRENT WINDOWS VERSION IS V8.4,
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* * * * * STN Columbus * * * * *

FILE 'HOME' ENTERED AT 15:11:21 ON 01 FEB 2010

=> file casreact		
COST IN U.S. DOLLARS	SINCE FILE	TOTAL
	ENTRY	SESSION
FULL ESTIMATED COST	0.66	0.66

FILE 'CASREACT' ENTERED AT 15:12:59 ON 01 FEB 2010

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FILE CONTENT:1840 - 30 Jan 2010 VOL 152 ISS 6

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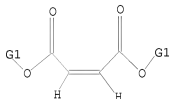
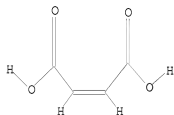
Uploading C:\Program Files\Stnexp\Queries\10577374-maleic.str

L1 STRUCTURE UPLOADED

=> d l1

L1 HAS NO ANSWERS

L1 STR



G1 Me,Et

Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 15:14:52 FILE 'CASREACT'

SCREENING COMPLETE - 102 REACTIONS TO VERIFY FROM

22 DOCUMENTS

100.0% DONE 102 VERIFIED 4 HIT RXNS 4 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED VERIFICATIONS: 1435 TO 2645
PROJECTED ANSWERS: 4 TO 199

L2 4 SEA SSS SAM L1 (4 REACTIONS)

=> s l1 full

FULL SEARCH INITIATED 15:14:57 FILE 'CASREACT'
SCREENING COMPLETE - 1677 REACTIONS TO VERIFY FROM 287 DOCUMENTS

100.0% DONE 1677 VERIFIED 34 HIT RXNS 28 DOCS
SEARCH TIME: 00.00.01

L3 28 SEA SSS FUL L1 (34 REACTIONS)

=> file caplus
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
128.66	129.32

FULL ESTIMATED COST

FILE 'CAPLUS' ENTERED AT 15:15:09 ON 01 FEB 2010
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FILE COVERS 1907 - 1 Feb 2010 VOL 152 ISS 6
FILE LAST UPDATED: 31 Jan 2010 (20100131/ED)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Dec 2009
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Oct 2009

CAPLUS now includes complete International Patent Classification (IPC) reclassification data for the third quarter of 2009.

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=> s l3

L4 28 L3

=> s l4 not py > 2004
7311664 PY > 2004

L5 17 L4 NOT PY > 2004

=> d 15 ibib abs 1-
YOU HAVE REQUESTED DATA FROM 17 ANSWERS - CONTINUE? Y/(N):y

L5 ANSWER 1 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN
ACCESSION NUMBER: 2005:396547 CAPLUS
DOCUMENT NUMBER: 144:369644
TITLE: Catalytic synthesis of dimethyl fumarate with
phosphotungstic acid
AUTHOR(S): Li, Yangshu; Yu, Bin
CORPORATE SOURCE: Science School, Nanjing University of Technology,
Nanjing, 210009, Peop. Rep. China
SOURCE: Huagong Shikan (2004), 18(2), 57-58
CODEN: HUSHT; ISSN: 1002-154X
PUBLISHER: Huagong Shikan Zazhishe
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
OTHER SOURCE(S): CASREACT 144:369644
AB Phosphotungstic acid was used as an esterification catalyst for
synthesizing di-Me fumarate (DMF), with maleic anhydride as the starting
material and potassium bromate KBrO3 as the isomerizing agent. This
method has the advantages of requiring small amount of catalyst with high
catalysis activity, resulting in shorter reaction time and high DMF yield
(typically over 90%). The purification procedure of DMF is simple.

L5 ANSWER 2 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN
ACCESSION NUMBER: 2005:230998 CAPLUS
DOCUMENT NUMBER: 143:442369
TITLE: Catalytic preparation of diethyl fumarate with sodium
acid sulfate
AUTHOR(S): Yang, Xin-bin
CORPORATE SOURCE: Department of Chemistry, Rongchang Branch of Southwest
Agricultural University, Chongqing, 402460, Peop. Rep.
China
SOURCE: Guangzhou Huaxue (2004), 29(4), 5-8
CODEN: GAHUFW; ISSN: 1009-220X
PUBLISHER: Zhongguo Kexueyuan Guangzhou Huaxue Yanjiuso
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
OTHER SOURCE(S): CASREACT 143:442369
AB Di-Et fumarate was prepared from fumaric acid and EtOH using NaHSO4 as
esterification catalyst. Under optimal esterification conditions: the
reaction temperature 130°, mol. ratio EtOH/fumaric acid = 4, the reaction
time 6 h, and the amount of NaHSO4 5% (based on fumaric acid), the yield of
di-Et fumarate was 93.5%. The method has the advantage of easy operation,
good yield, and the catalyst could be reused.

L5 ANSWER 3 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN
ACCESSION NUMBER: 2004:795860 CAPLUS
DOCUMENT NUMBER: 142:431894
TITLE: Study on the new technology of synthesis of dimethyl
fumarate
AUTHOR(S): Fan, Guo-zhi
CORPORATE SOURCE: Biological and Chemical Engineering Department, Wuhan
Polytechnic University, Wuhan, 430023, Peop. Rep.
China
SOURCE: Yingyong Huagong (2004), 33(3), 44-46
CODEN: YHUA7; ISSN: 1671-3206
PUBLISHER: Yingyong Huagong Bianjibu
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
OTHER SOURCE(S): CASREACT 142:431894
AB Di-Me fumarate was prepared by esterification using H3PW12O40/C as catalyst.
Under optimum conditions, the product. yield reached 88.1%.

L5 ANSWER 4 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN
 ACCESSION NUMBER: 2003:44804 CAPLUS
 DOCUMENT NUMBER: 138:337689
 TITLE: Synthesis of dimethyl fumarate by heterogeneous supported heteropoly acid
 AUTHOR(S): Xu, Wenyuan; Peng, Daofeng; Xiong, Guoxuan; Zhu, Xiaping
 CORPORATE SOURCE: Department of Applied Chemistry, East China Institute of Technology, Fuzhou, 344000, Peop. Rep. China
 SOURCE: Huaxue Shiji (2002), 24(6), 367-368
 CODEN: HUSHDR; ISSN: 0258-3283
 PUBLISHER: Huagongbu Huaxue Shiji Xinsizhan
 DOCUMENT TYPE: Journal
 LANGUAGE: Chinese
 OTHER SOURCE(S): CASREACT 138:337689
 AB Synthesis of di-Me fumarate by esterification reaction of fumaric acid with methanol catalyzed by heterogeneous supported heteropoly acid PW12/C was studied in this paper. A careful study of the effects on the esterification reaction was done. Under these conditions, the yield of ester was about 91.6%.

L5 ANSWER 5 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN
 ACCESSION NUMBER: 2002:952405 CAPLUS
 DOCUMENT NUMBER: 139:6591
 TITLE: Catalytic synthesis of dimethyl fumarate using solid-supported superacid catalyst
 AUTHOR(S): Zhao, Lifang; He, Zhusheng; Ma, Yuying
 CORPORATE SOURCE: Dept. Chem. + Chem. Eng., Baoji Coll. Arts + Sci., Baoji, 721007, Peop. Rep. China
 SOURCE: Baoji Wenli Xueyuan Xuebao, Ziran Kexueban (2002), 22(2), 138-140
 CODEN: BWZKFL
 PUBLISHER: Baoji Wenli Xueyuan Xuebao Bianjibu
 DOCUMENT TYPE: Journal
 LANGUAGE: Chinese
 OTHER SOURCE(S): CASREACT 139:6591
 AB The preparation of supported catalyst, TiO2/La3+/SO42- supported on mol. sieves, and its catalytic activity to esterification of fumarate were studied. The catalyst had fine catalytic activity. The optimum conditions of the esterification were decided by orthogonal expts. as follows: activation temperature of the catalyst was 500°, the amount of catalyst was 15% (based on the mass of fumaric acid), the mole ratio of alc. to acid was 6:1 and the reaction time was 5 h. Under the optimum reaction conditions, the yield of di-Me fumarate was up to 92.3%.

L5 ANSWER 6 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN
 ACCESSION NUMBER: 2002:903288 CAPLUS
 DOCUMENT NUMBER: 138:271016
 TITLE: A simple, convenient and expeditious route to methyl esters of carboxylic acids by thionyl chloride-methanol
 AUTHOR(S): Chatterjee, Tapasi; Chattopadhyay, Subhagata
 CORPORATE SOURCE: Department of Chemistry, Jadavpur University, Kolkata, 700 032, India
 SOURCE: Oriental Journal of Chemistry (2002), 18(2), 187-190
 CODEN: OJCHEG; ISSN: 0970-020X
 PUBLISHER: Oriental Scientific Publishing Co.
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 138:271016
 AB A simple, convenient and expeditious preparation of 40-90% Me esters of carboxylic acids by thionyl chloride and MeOH was described. Among the 29

esters prepared were 90% 2-IC6H4CO2Me, 87% 4-MeOC6H4CO2Me and 86% Bz(CH2)2CO2Me.

REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 7 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN

ACCESSION NUMBER: 2002:81290 CAPLUS

DOCUMENT NUMBER: 137:352688

TITLE: Catalytic reaction-distillation synthesis of dimethyl fumarate by fixed-carried heteropoly acid

AUTHOR(S): Ding, Bin; Guo, Xiangming

CORPORATE SOURCE: Jilin Institute of Chemical Technology, Jilin, 1320022, Peop. Rep. China

SOURCE: Dongbei Shida Xuebao, Ziran Kexueban (2001), 33(4), 61-65

CODEN: DSZKEE; ISSN: 1000-1832

PUBLISHER: Dongbei Shifan Daxue Xueshu Qikanshe

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

OTHER SOURCE(S): CASREACT 137:352688

AB A new synthesis technol. of di-Me fumarate was presented. Fumarate, methanol, and self-made fixed-carried heteropoly acid as catalyst were used. The reaction-distillation conditions were ratio of alc. and acid about 7:1; esterification temperature about 67-78°; and reaction time ≤6 h. The yield of product was up to 92%.

OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD (1 CITINGS)

L5 ANSWER 8 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN

ACCESSION NUMBER: 2001:535025 CAPLUS

DOCUMENT NUMBER: 136:294520

TITLE: Synthesis of dimethyl fumarate catalyzed by

SO42-/TiO2/La3+ rare earth solid superacid

Zhou, Jianwei

CORPORATE SOURCE: Department of Chemical Engineering, Pingyuan University, Xinxiang, 453003, Peop. Rep. China

SOURCE: Henan Huagong (2001), (5), 12-14

CODEN: HEHUF3; ISSN: 1003-3467

PUBLISHER: Henansheng Shiyou Huaxue Gongye Keji Qingbao Zhongxinzhuan

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

OTHER SOURCE(S): CASREACT 136:294520

AB Di-Me fumarate was synthesized from fumaric acid and methanol with SO42-/TiO2/La3+ rare earth solid superacid as catalyst in dichloromethane solvent. Optimum synthetic conditions were determined: molar ratio of fumaric acid to methanol 6:1, dosage of catalyst 1.0 g/0.1 mol fumaric acid, time 4 h and solvent 25 mL. Yield of product reached above 94%.

L5 ANSWER 9 OF 17 CAPLUS COPYRIGHT 2010 ACS ON STN

ACCESSION NUMBER: 2001:461290 CAPLUS

DOCUMENT NUMBER: 136:279092

TITLE: Synthesis of dimethyl fumarate from maleic acid

Cao, Kelin

CORPORATE SOURCE: Shanxi Taiming Chemical Engineering Co., Ltd., Taigu, 030800, Peop. Rep. China

SOURCE: Huagong Jinzhan (2001), 20(4), 33-34, 39

CODEN: HUJIEK; ISSN: 1000-6613

PUBLISHER: Huaxue Gongye Chubanshe

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

OTHER SOURCE(S): CASREACT 136:279092

AB Title compound was prepared from maleic acid, isomerized fumaric acid in the

presence of ammonium persulfate as catalyst, further esterification with methanol in the presence of phosphotungstic acid as catalyst, giving product with yield over 94%. The effects of catalysts and catalyst amount on the reactions were studied.

L5 ANSWER 10 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 2000:832322 CAPLUS

DOCUMENT NUMBER: 134:310893

TITLE: Synthesis of dimethyl fumarate catalyzed by composite solid superacid SO₄2-/TiO₂-Al₂O₃

AUTHOR(S): Cheng, Yonghao

CORPORATE SOURCE: Department of Chemistry, Hebei Normal University, Shijiazhuang, 050016, Peop. Rep. China

SOURCE: Riyong Huaxue Gongye (2000), 30(5), 12-13

CODEN: RHGOE8; ISSN: 1001-1803

PUBLISHER: Qinggongyebu Kexue Jishu Qingbao Yanjiusuo

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

OTHER SOURCE(S): CASREACT 134:310893

AB Di-Me fumarate was synthesized from fumaric acid and methanol with composite solid superacid SO₄2-/TiO₂-Al₂O₃ as catalyst. Optimum synthetic conditions were determined: methanol:fumaric acid 6:1, time 4 h, and dosage of catalyst 3 g. Yield of product reached 91.4%.

L5 ANSWER 11 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1994:269626 CAPLUS

DOCUMENT NUMBER: 120:269626

ORIGINAL REFERENCE NO.: 120:47747a, 47750a

TITLE: Catalytic synthesis of dimethyl fumarate with ferric chloride

AUTHOR(S): Yu, Shanxin; Lei, Huanwen

CORPORATE SOURCE: Dep. Chem., Hunan Norm. Univ., Changsha, 410081, Peop. Rep. China

SOURCE: Huaxue Shiji (1993), 15(6), 374, 376

CODEN: HUSHDR; ISSN: 0258-3283

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

OTHER SOURCE(S): CASREACT 120:269626

AB Ferric chloride (FeCl₃·6H₂O) can be used as a catalyst for the esterification reaction of fumaric acid instead of sulfuric acid. The conditions in synthesis of di-Me fumarate catalyzed with FeCl₃·6H₂O are described. The advantages of this method are: simple procedure, mild reaction conditions, non-corrosive, less pollution and purer product.

L5 ANSWER 12 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1990:493372 CAPLUS

DOCUMENT NUMBER: 113:93372

ORIGINAL REFERENCE NO.: 113:15639a, 15642a

TITLE: Chemical evolution of the citric acid cycle: sunlight and ultraviolet photolysis of cycle intermediates

AUTHOR(S): Waddell, Thomas G.; Geevarghese, Sunil K.; Henderson, Barry S.; Pagni, Richard M.; Newton, Jessica S.

CORPORATE SOURCE: Dep. Chem., Univ. Tennessee, Chattanooga, TN, 37403, USA

SOURCE: Origins of Life and Evolution of the Biosphere (1989), 19(6), 603-7

CODEN: OLEBEM; ISSN: 0169-6149

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 113:93372

AB Sunlight or laboratory UV photolyses of oxalacetic, succinic, fumaric, malic, and citric acids were carried out at 0.1M aqueous solns. The nonvolatile products were isolated and identified by gas chromatog./mass spectroscopic

anal. of derived Me esters. Several conversions corresponding to modern citric acid cycle reactions were observed. Notably, oxalacetic acid gave citric acid as the major product of sunlight photolysis. Other identified products relate to chemical evolution and further support the important role of succinic acid in the origin of life.

OS.CITING REF COUNT: 2 THERE ARE 2 CAPLUS RECORDS THAT CITE THIS RECORD
(2 CITINGS)

L5 ANSWER 13 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1986:167832 CAPLUS
DOCUMENT NUMBER: 104:167832
ORIGINAL REFERENCE NO.: 104:26571a,26574a
TITLE: Photooxidative cleavage of catechol
AUTHOR(S): Liu, Zhujin; Yu, Qiansheng
CORPORATE SOURCE: Shanghai Inst. Org. Chem., Acad. Sin., Shanghai, Peop.
Rep. China
SOURCE: Huaxue Xuebao (1985), 43(11), 1110-13
CODEN: HHHFA4; ISSN: 0567-7351
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
OTHER SOURCE(S): CASREACT 104:167832
AB Photooxidative cleavage of catechol gave fumaric acid and glyoxalic acid hydrate. The mechanism was discussed.

L5 ANSWER 14 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1983:452585 CAPLUS
DOCUMENT NUMBER: 99:52585
ORIGINAL REFERENCE NO.: 99:8211a,8212a
TITLE: Reactions of cyclic anhydrides. Part IX. Facile esterification of carboxylic acids with organophosphorus reagents. Novel application of alkylphosphoric esters (APE)
AUTHOR(S): Balasubramanian, V.; Bhatia, V. G.; Wagh, S. B.
CORPORATE SOURCE: Sci. Res. Cent., H.P.T. Arts and R.Y.K. Sci. Coll., Nasik, 422 005, India
SOURCE: Tetrahedron (1983), 39(9), 1475-85
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 99:52585
AB The APE reagent, prepared from P4010 and excess alkanol, was used for the esterification of carboxylic acids (.apprx.50), including maleanilic, fumaranilic, and succinanilic acids.
OS.CITING REF COUNT: 8 THERE ARE 8 CAPLUS RECORDS THAT CITE THIS RECORD
(8 CITINGS)

L5 ANSWER 15 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN

ACCESSION NUMBER: 1982:217212 CAPLUS
DOCUMENT NUMBER: 96:217212
ORIGINAL REFERENCE NO.: 96:35877a,35880a
TITLE: Orthoamides. XXXVIII. Chemistry of orthocarbonic acid esters and α,α,α -trialkoxycetonitriles
AUTHOR(S): Kantlehner, Willi; Maier, Thomas; Loeffler, Wolfgang; Kapassakalidis, Joannis J.
CORPORATE SOURCE: Inst. Org. Chem. Biochem. Isotopenforsch., Univ. Stuttgart, Stuttgart, D-7000/80, Fed. Rep. Ger.
SOURCE: Liebigs Annalen der Chemie (1982), (3), 507-29
CODEN: LACHDL; ISSN: 0170-2041
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 96:217212
AB The reactions of (EtO)4C with carboxylic acids and anhydrides, alcs.,

diols, anilines, amines and their HCl salts, cyclic imides, hydrazides, imidazoles, and amino acids were given and discussed. Also studied were the reactions of (EtO)3CCN with alcs., Na alkoxides, phenols, and amines.

OS.CITING REF COUNT: 4 THERE ARE 4 CAPLUS RECORDS THAT CITE THIS RECORD (4 CITINGS)

L5 ANSWER 16 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN
ACCESSION NUMBER: 1971:510027 CAPLUS
DOCUMENT NUMBER: 75:110027
ORIGINAL REFERENCE NO.: 75:17371a,17374a
TITLE: Convenient method of esterification of unsaturated organic acids using a boron trifluoride etherate-alcohol reagent
AUTHOR(S): Kadaba, Pankaja K.
CORPORATE SOURCE: Coll. Pharm., Univ. Kentucky, Lexington, KY, USA
SOURCE: Synthesis (1971), (6), 316-17
CODEN: SYNTBF; ISSN: 0039-7881
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 75:110027

AB A BF3.OEt-alc. reagent was used to directly and selectively esterify unsatd. carboxylic acids. A mixture of 0.1 mole acid, 0.1 or 0.2 mole BF3.OEt (depending on the number of carboxyl groups in the acid), and the appropriate alc. (a 10-fold excess relative to BF3.OEt) was refluxed for 24 hr to yield the esters. Both aliphatic and aralkanoic acids were esterified.

OS.CITING REF COUNT: 6 THERE ARE 6 CAPLUS RECORDS THAT CITE THIS RECORD (6 CITINGS)

L5 ANSWER 17 OF 17 CAPLUS COPYRIGHT 2010 ACS on STN
ACCESSION NUMBER: 1958:103785 CAPLUS
DOCUMENT NUMBER: 52:103785
ORIGINAL REFERENCE NO.: 52:181961,18197a-c
TITLE: Esterification with trapping phase
AUTHOR(S): Klostergaard, Henry
CORPORATE SOURCE: California Inst. Technol., Pasadena
SOURCE: Journal of Organic Chemistry (1958), 23, 108-10
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 52:103785

AB The problem involved in the synthesis of an ester with a higher b.p. than the component acid or alc. was solved simply by a procedure based on phase separation. An equivalent of acid and 1.25-1.33 equivs. alc. refluxed 15 min.

with a catalytic amount of concentrated H2SO4 and the solution diluted under continuing reflux with a volume of PhMe equal to that of the expected ester, the mixture treated gradually with 1 ml. concentrated H2SO4 for each ml. H2O present and formed, the PhMe layer removed, and the residual phase refluxed 5 min. with 25% of the previously used amount of PhMe, the PhMe layer removed and the process repeated, the combined PhMe exts. washed with H2O, and the dried (Na2SO4) extract fractionated gave 80, 80, 80, 83, 72, and 85% yields of the Et esters of (CO2H).2H2O, b. 66°, 76°, 84° at 5, 10, 15 mm., citric acid monohydrate (I), b. 164°, 177°, 183° at 5, 10, 15 mm., adipic acid (II), b. 111° 125°, 133° at 5, 10, 15 mm., succinic acid (III), b. 88°, 99°, 106° at 5, 10, 15 mm., furoic acid (IV), b. 90°/15 mm., and levulinic acid (V), b. 80°, 90°, 96° at 5, 10, 15 mm. Certain simplifications were found to be possible. A similar procedure using CaCl2 and 38% HCl yielded 64, 89, 92, 72.93, 94, 68, and 89% Et esters of aconitic acid, b. 150° 160°, 170° at 5, 10, 15 mm., fumaric acid, b. 87°,

98°, 106° at 5, 10, 15 mm., maleic acid, b. 86°,
99°, 106° at 5, 10, 15 mm., I, III, II, IV, and V, resp.

Oxalic and tartaric acids were not esterified by this later method and
neither procedure succeeded with tartaric acid.

OS.CITING REF COUNT: 1 THERE ARE 1 CAPLUS RECORDS THAT CITE THIS RECORD
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